BEST AVAILABLE COPY

PATENT

I hereby certify that this paper or fee is being transmitted sufficient postage as Express Mail No. EV 531802399 US, addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on January 14, 2005.

Dated: January 14, 2005 BY:______________________
Rodney D. DeKruif

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:	David C. Dunand	)
		)
Serial No:	10/680,639	)
		) Attorney Docket No. 6513-DIV
		)
Filed:	October 7, 2003	)
		)
For:	SUPERCONDUCTING Mg-MgB2 AND RELATED METAL COMPOSITES AND METHODS OF PREPARATION	)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

RULE 131 DECLARATION OF DAVID C. DUNAND

1. I, David C. Dunand, am inventor of the invention (the "Invention") disclosed and claimed in the above-entitled application (the "Application"). I am a Professor in the Department of Materials Science and Engineering at Northwestern University. I make this declaration in support of the Application and, in particular, to antedate a reference cited against the Application.

2. The Invention claimed in the Application was completed before the effective date of the journal article by Sharoni, et al., entitled "Spatial variations of the superconductor gap structure in MgB_2/Al composite," *J. Phys. Condens. Matter* 13 (2001) L503-L508 (*i.e.*, the Sharoni reference). More specifically, the

Invention was conceived and with due diligence reduced to practice prior to the effective date of the Sharoni reference. (The effective date of publication, i.e., June 4, 2001, can be found at http://www.iop.org/EJ/toc/0953-8984/13/22.)

3. This Declaration, and prior invention, is supported by copies of pertinent pages from my laboratory research notebook, entries which were contemporaneously witnessed by Graduate Student Naomi Davis. Date redacted copies of the aforementioned notebook pages are provided collectively as Exhibit A and incorporated herein by reference. These documents establish that the Invention was made at least as early as March 26, 2001, which is a date earlier than the effective date of the Sharoni reference.

I hereby declare that: All statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; that those statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code; and that willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

Date January 13, 2004

David C. Dunand

Monday

I discussed last week with D.N. Seidman the idea of superconducting composites based on the new compound MgB_2. I had mentioned to him the following simple idea:

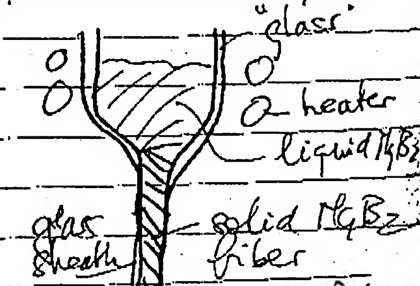
① Infiltrated Mg-MgB_2 composites

The Mg-B phase diagram shows that Mg and MgB_2 are at equilibrium with each other. It should thus be possible to infiltrate a packed bed of MgB_2 powder (which may have been pre-sintered to make a continuous skeleton) with liquid Mg. The net result is a composite with a continous superconducting phase of MgB_2 embedded in a continuous metallic Mg phase, which allows for thermal management (conducting heat away from MgB_2 and preventing loss of superconductivity) and also being able to carry current if s.c. is lost.

The following other ideas came to me, undisclosed to DN Seidman:

② Fabrication of MgB_2 fibers

Superconducting fibers would be very useful embedded in a non s.c. matrix (polymer, metal, glass, ceramic) which provides thermal and mechanical properties. Existing s.c. fibers can be made by drawing (when ductile) or by powder sintering (when brittle). My idea uses a different technique, i.e. the Taylor wire technique. MgB_2 powder are packed in a "glass" tube (any amorphous ceramic such as Pyrex, E glass, etc). The MgB_2 is melted and the glass is quickly drawn into a hollow fiber containing MgB_2 which solidifies into a continuous fiber, which can be spooled



Hasomu J. Valn

Read & understood NDS

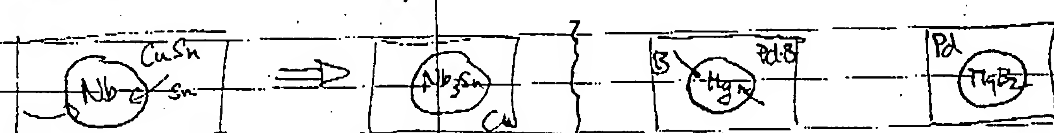
Exhibit A

Taylor wires have been made with many metals (see Donald, 1987). The following requirements are needed: (1) no reaction between glass and MgB_2, (2) working temperature of glass must be higher than MgB_2 (3) glass must become highly viscous before MgB_2 solidifies, lest it deforms and breaks the MgB_2. Pyrex seems a good first choice.

The glass sheathed MgB_2 fiber can then be embedded in a matrix (metal, glass, ceramic, polymer) to form a composite by usual composite processing techniques. Alternatively, the glass can be dissolved by an acid (HF-containing acid) and the bare MgB_2 fibers can be used in a composite. Some dissolution of MgB_2 may be tolerable as part of the glass removal.

③ Ductile Mg-B-Pd wires as precursors for Pd-MgB_2 composites.

Nb_3Sn-Cu composites are ideal, as Cu prevents thermal run-away. They are produced by stacking Nb wires between a Cu-Sn bronze matrix and drawing, resulting in a ductile Cu-Sn / Nb composite which can be drawn and shaped (coiled, etc) to shape. Heat-treatment diffuses Sn to the Nb fibers, forming the brittle Nb_3Sn superconductor in a Cu matrix



The same idea can be used for MgB_2 - X composites, where X is a ductile metal which can be alloyed with B. A good example is Pd, which can accept 2.2 wt% B (at%) So, Mg wires could be embedded within sheets of Pd-B alloy, diffusion-bonded, drawn or shaped as needed. Upon heating below 650°c (melting point of Mg), ... MgB_2 ... leaving behind pure Pd

calculations for volume fractions of Pd/MgB_2 composite

- MgB_2 synthesis $\quad Mg + 2B \rightarrow MgB_2$

(24g/mol) (10.8g/mol) (45 g/mol)

$\overset{24.3}{24g}$ + 21.6 g → 45.6 g

(1.74g/cc) (2.34 g/cc) (2.63 g/cc)

$\frac{9.23}{13.97+9.23} = 0.398$ $\quad \overset{13.97}{13.8}$ cc + 9.23 cc → 17.11 cc

$\frac{9.23}{17.11} = 0.539$ $\quad$ shrinkage: 5.9 cc (27%)

- ~~Pd~~-B alloy $\quad$ ~~Pd~~-2.2wt% B (max solubility) $\quad$ e.g.

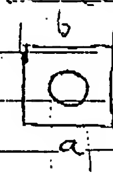
21.6 g B → 960.2 g Pd

- Mg-B-Pd alloy

960.2 g Pd (12g/cc) → 80.0 cc Pd

45.6 g MgB_2 → 17.11 cc MgB_2

$\boxed{\begin{array}{l}83.4\text{ vol\% Pd}\\ 17.6\text{ vol\% }MgB_2\end{array}}$ Mg



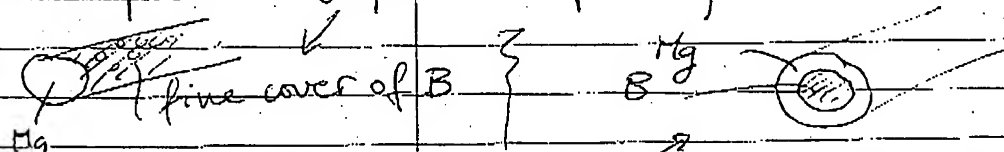
→ 1/6 of the volume is MgB_2

$\frac{\pi a^2}{4b^2} = 0.176 \qquad \frac{a}{b} = \sqrt{0.176\cdot\frac{4}{\pi}}$

This volume fraction is not very high, but still commercially feasible, provided price of Pd is not too high

. NB.: some Mg will dissolve in Pd

- Additional B could be introduced by dipping Mg wires in B suspension before compacting with Pd or Pd-B foils

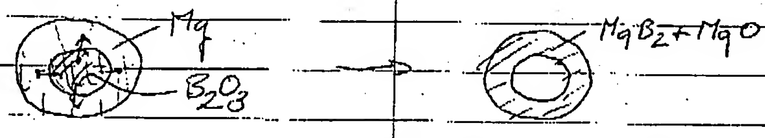


or filling Mg tubes with B powder and then compacting with Pd. Extrusion would still be easy with B powder

read + understood $\quad$ Marvin G. [illegible]

- Alternative is to fill Mg tubes with B_2O_3, which is viscous at extrusion temperatures. The ~~mat~~ reaction is then

$4Mg + B_2O_3 \rightarrow MgB_2 + 3MgO$



or a suspension of B particles in B_2O_3, which will remain viscous & drawable.

e.g. $5Mg + (B_2O_3 + 2B) \rightarrow 2MgB_2 + 3MgO$

The advantage is that the metallic matrix can now be a metal different from Pd, and the volume fraction of fibers has no restrictions

MgB_2 fibers (160 μm in ϕ) have been disclosed at the APS March meeting: B fiber is exposed to Mg vapors and form MgB_2 which was found to be superconducting.

Taylor wire technique has following advantages:
- much more rapid production
- diameter easily changed (1-100 μm typically)
- different grain structure
- sheath of glass.

good matrices for "filled-tube" process: any metal which does not dissolve into Mg (which would contaminate MgB_2)
1. Cu: zero sol. in Mg; eutectic at 487°C; $MgCu_2$ & Mg_2Cu; ~2wt% Mg in Cu
2. Ni: zero ... 506°C, $MgNi_2$ & Mg_2Ni; zero

read & understood, [signature]

Solubilities

Metal X	X in Mg	Mg in X	Intermetallics	Eutectic (C)
Cu	0	~3wt%	2	483
Ni	0	0	2	506
Ti	0.12	~1.5at%	0	651
Co	0	0	1	635
Fe	0	0	0	—
Cr	0	0	0	—
Au	0	huge	many	575
Be	0	0	1	—
Pd	0.23	5wt%	7	540
Pt	0	0 ?	5	575
Nb	0	0	0	—
Mo	0	0	0	—

Other advantages:

high conductivity: Cu (but reduced by Mg in solid soln)
Au (but β)

Processing: Cu low melting
electrodeposition: Cu, Ni, Cr, Au, Pd?, Pt?

no bonding: Cu, Au

read & understood
[signature]

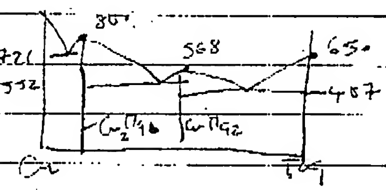
JAN-12-2005 05:15PM FROM- T-305 P.006 F-070

Other Idea:

React B with Cu-Mg melt $2B + Cu + Mg \rightarrow MgB_2 + Cu$

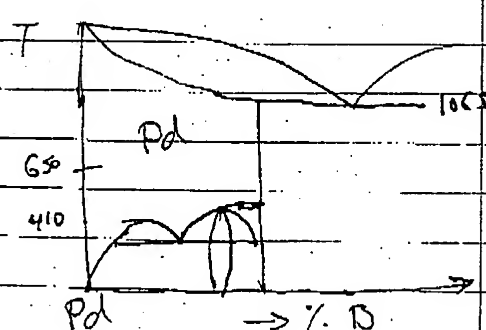
this is only possible if Cu does not dissolve and deteriorates MgB_2 s.c. properties

1. Infiltrate B fibers or B skeleton with Cu-Mg melt
2. react in the melt
3. solidify Cu - MgB_2 composite.



This will work with non-boride formers, i.e. mostly Cu and Au

Read & Understood



pure B dissolves Cu (~ 16 wt%) at 1013°C

1 mol B = 4.61 cm³ ~ 40 vol%

1/2 mol Mg = 6.89 cm³ ~ 60 vol%

30 vol% B } ~ 50 vol% MgB_2 → 2/3 MgB_2
45 vol% Mg
25 vol% Cu 25 vol% 1/3 Cu

?Pd?

Dorian

BMG → batch program works! → code
some cryst. matrix! → new paper with double peaks
no creep, error due to reloading after
≥ 1h hold → throw away data

stand alone paper { – compression
– hold
– matrix info

• MRS paper → draft on Monday
practice talk on Thursday

•

TaskerCarr Dearborn
↳ ↳ ~~st~~ carbon steel

Taylor wire MgB_2 trial

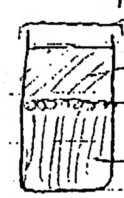
Went to glass blowing shop and tried with pyrex (10 mm)
– can draw easily empty wire
– MgB_2 powder (~ 1cm high) gets red hot, but no melting visible
Can partially draw pyrex, but eventually fracture. MgB_2 does not burn.
Problem is ϕ is too large, cannot keep the whole block of MgB_2 hot. → try finer tube, maybe try a torch with multiple outlets.

Tried finer wires ~ 4 mm, and after a few trials, got a wire ~ 100-200 μm, with black MgB_2 inside. It is not clear whether the powder was melted throughout, but it seemed to be powder

- Tried Cu in 4 mm tube (pyrex), but not successful due to high T_m (glass necked before Cu melted)
- Zn powders worked, nice Taylor wires.
- Tried again MgB_2 and got a nice length ~ 10 cm uninterrupted. Pyrex was not cleaned, so bubbles interrup wire in some parts. Maybe also need to "premelt" materials, e.g. as done for AgCl during thesis (but $T_m = 455°C$ vs $800°C$ for MgB_2)

glass shop closed
read Canfield paper (PRL 86, 2423) and had the following idea: integrated fiber formation & composite fabrication

1. Put B fibers and Mg into crucible
2. Heat at 950°C ⇒ Mg melts and seals crucible
   hold for 2h ⇒ Mg vapors react with B fibers and form MgB_2 fibers like Canfield's paper



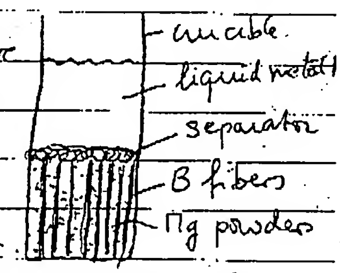
3. Pressurize with gas and force Mg into fiber preform
   → MgB_2 – Mg Composite

One of the main advantages is that MgB_2 fibers are formed in-situ. They do not have to be handled, bundled, etc, which would easily damage or break them, because Canfield describes them

Another possibility is to infiltrate B-fibers with molten Mg and keep the composite at 950°C for 1-2 h, long enough for complete reaction to form MgB_2. Solidifying gives a Mg composite with MgB_2 fibers.

Method to produce MgB_2 metal matrix composites

Canfield's paper shows that MgB_2 fibers can be produced by exposing B fibers to Mg vapors at 950°C for 2h. However, the fibers are very brittle and bent after fabrication. It will be difficult to bundle them and subsequently infiltrate them without breakage. A solution is to synthesize and infiltrate the fibers ~~and~~ in two closely consecutive steps, without handling the fibers.

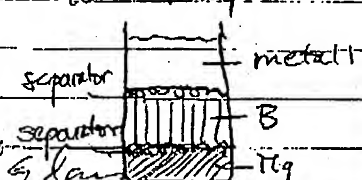


Step 1. Heat-up assembly to synthesis temperature. Mg melts and vaporizes, reacting with B fibers to form MgB_2. If the metal 1 is melted, it forms a liquid seal, thus preventing escape of Mg vapors. A non-wetting separator prevents metal 1 from contacting fibers. Examples for metal 1: Mg, Al, Cu alloy with low ~~[illegible]~~ solidus (bronze, brass)

Step 2: pressurize with gas the crucible, forcing liquid metal 1 through the separator and between the MgB_2 fibers, thus making a composite. Solidify and extract composite from crucible. If metal 1 has higher melting point than synthesis temperature of fibers, first raise temperature to melt it.

Complex shapes could be made by this process.

A variation is to put Mg below fibers with separator permeable to Mg vapors.



read + understood [signature]

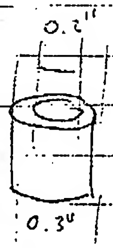
see p136: 24 g ~~B~~ Mg   13.8 cc Mg   60 vol %

+ 21.6 g B   9.23 cc B   ~ 40 vol %

so at best: 20 vol % B

30 vol % Mg powder

50 vol % porosity



ϕ 5 mm $A = 19.6\ mm^2 \rightarrow 3.9\ mm^2$ B

0.2"

each fiber is 100 μm $\phi \rightarrow A$ $\pi \cdot 0.05^2 =$ $0.2 \cdot \left(\frac{5}{0.1}\right)^2$ 500 fibers

@ 2 cm → 10 m

drop into 0.3" crucible.

Observe Dorian run infiltration

1. Turn H_2O on (circulate first at < 125 psi)
   open to machine → flow switch on
   zero pressure transducer
2. Close vessel - slide in
   - put Pb sheet
   - tighten nut until lid is flush with vessel
3. Connect 1 p-transducer (showing as psi x 5, full vac = -65/70
   4 TC (check then
4. Open gas tank, set 2nd stage regulator at 150 psi
5. purge 3 times - use valve 1 only
6. check for vac. leak: insolate vessel
   verify vac. is constant for 4-5 min
7. Switch on main power, turn to 50% on both variacs
   → upper zone: 5.5 A / 100 V
   → lower zone: 4 A / 100 V

   wait ~1½ h → 350°C
8. put T recorder on — turn to 75% (top only)
   → upper zone: 8.5 A / 150 V

   wait ~ 45 mins → turn to 90% (top only) 70% bottom
   wait ~ 40 min → turn down to 50% (both) and fine-tune

   45 mins
9. Infiltrate
   1. increase to 550 psi (regulator)
   2. open valve #2 to vessel
   3. switch off power
   4. open valve #1 and pressurize to 500 psi (35 atm)
   5. close valves 1 & 2 (will leak ~ 1-2 psi/min)
10. Cool down ~ h to 200°C
   turn off water, vent (use vent valve) open

Mg 99.99% 2 pieces from Emma (26.53g)
started cutting pieces
cut tubing & cleaned tubing

{ tubes: 0.555" ID = 14.10mm
  0.305" ID = 7.75 mm

filling in small tube to 2cm : $V = 943\ mm^3 = 0.943\ cm^3 \rightarrow$
assuming 50% packing : $0.472\ cm^3$

$0.472\ cm^3$ ~~•~~ 2.63 g/cc $\rightarrow$ 1.24 g MgB_2
1.70 $\rightarrow$ 0.802 g Mg $\xrightarrow{\text{double}}$ 1.60 g Mg

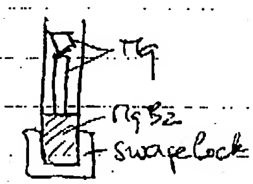
for large tube, multiply by $\left(\frac{0.555}{0.305}\right)^2 = 3.3 \rightarrow \boxed{\begin{array}{l}4.1g\ MgB_2\\ 5.3g\ Mg\end{array}}$

(called by ( )

She will send ~ 30 feet of 4 mil B fibers for free
Goodfellow charges ~ \$100 for 10 m. [minimum order 1.16 \$992 for 4 mil, \$1159 for 6 mil
5 μm W core, 100 μm diameter, 20.2 mg/m
$\Rightarrow$ 10 m = 0.202 g ...

prepare small crucible: Mg: 1.605 g (2 pieces)
add MgB_2 to crucible bottom tar: ~~47~~.210 g
-629 top + push with rod
48.208
383
48.441g
$\hookrightarrow$ 1.231g MgB_2

add pieces of Mg on top



measured height: 6.5 mm 22
not right, was not "touching bottom" at first!

4.5 mm for 0.233 g $\Rightarrow$ ~ 22.5 mm for full mass, OK!
leave for evening

Sue Abkowitz

modulus measurement

	RT E(Msi)	600°F G(Msi)
Ti64	: 16.88	
Ti64+10W	: 15.67 ↳ (5.609) −10%	5.041 → 5.052 → 5.056 → 5.060 → 5.067
		0 5 min 10 15 20 min

$\frac{0.02}{5.04}$ change 0.4% in 20 min

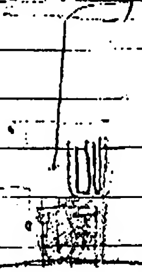
- W dissolution : no, T too low, G increases
- W form of : no, " " (check if Ar is used)
- β → α transf. is most likely!

mass : 4.633
volume $\frac{\pi 1.41^2}{4} \cdot 1.5 = 2.34 cm^3$
↳ 1.98 g/cm³

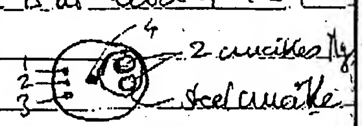
prepare large crucible   4.633 g MgB_2 (Alfa Aesar) (15 mm height)
5.768 g Mg −99.99% (2 large pieces, small)

- put a thin ZrO_2 felt layer on top of Mg, stuff crucible to the top w. Al_2O_3 felt (to prevent powder blow out during rx)
- load sample in vessel, contained in S.S. crucible/pot
- evacuate/purge 3 times
- 11:35 vacuum −76, insulate vessel (leak check)

	up	low	T	T2	T3	T4	vac
12:15							−75 → ok, no leaks
12:18	50%	50%				20.7	−76
12:30	60%	60%				191	−73
1:40	"	"				510	−70
2:00	75%	75%	582			568	−70
2:35	65%	65%	742	743	743	734	−69
2:43			735	737	739	735	
2:49	68%	68%				732	
2:55	"	"	734	737	739	734	switch off power

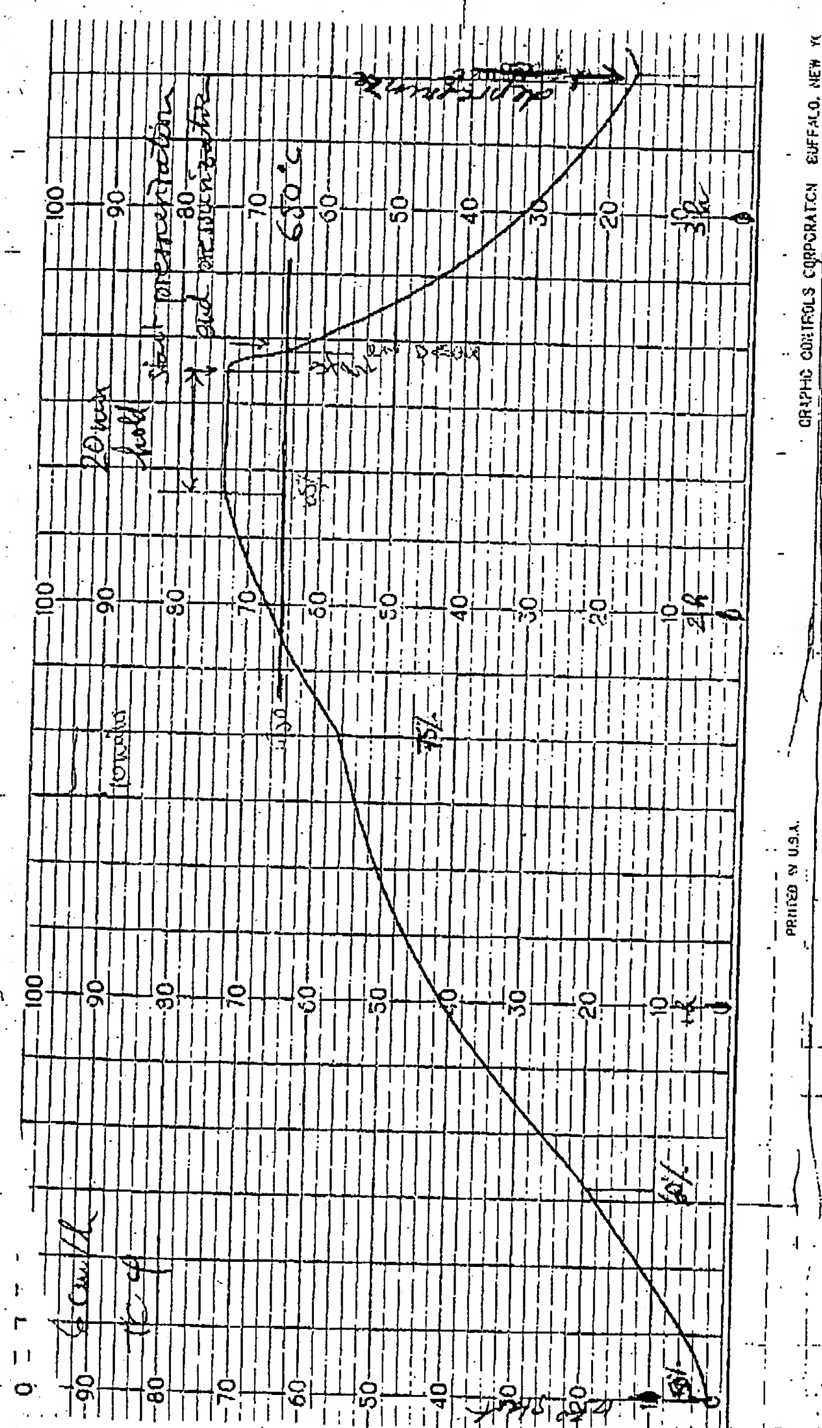


Mg is at level of TC4



2 minute pressurization to 2400 (980 psi)
3:40 depressurize 1140°C, 1400 (280 psi)

ent
-067
20 mi
20 min
wed)
$= 2.3 cm^2$
g/cm^3
bt)
and)
ble
way...)
TC4
ucibles Mg
awake



- open at ~ 160°C
- crucibles are fine, insulation is on top
- remove insulation Mg was melted, but level seems high → did Mg make a pool & seal?

- cut open large crucible: pieces were not melted! except for bottom piece which ~~showed minor~~ slumping. The time held and/or max temp. were too low!

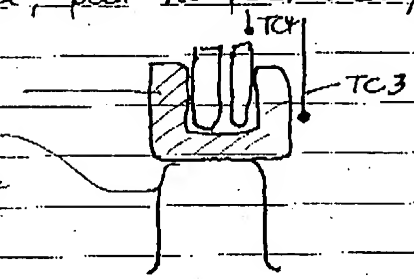
- pour MgB_2 powder on paper → reuse
- reuse Mg 2 pieces, except cut off top which was in contact with ZrO_2, looks very black.
- Some white deposit in tube above Mg, possibly as reaction with ZrO_2?

—·· 030
939

new large crucible: old $Mg\,B_2$ powder from previous expt 3.475g
new 0.939
$\boxed{4.414g}$ (Alfa Aesar)

old Mg: 4.558g 2 pieces
new Mg: 2.4704 1 piece
$\boxed{7.028\,g}$ (99.99%)

add ~~0.19g~~ 0.307g Mg to small crucible
both crucibles have Mg to the rim, put no felt on top
• load crucible in infiltrator
put them in the graphite crucible
on top of s.s. crucible
much less insulation as last time
should get better heating
~~TC3 is next to gr. crucible~~
TC4 is just above steel crucibles



• evacuate to −67, insulate & leave for the night 11:35 PM

9³⁰ AM, vac at −41, good seal! Evacuate & flush twice

	P	T_1	T_2	T_3	T_4	upper	lower	
9:55	−55	24	22	19	21	70%	70%	change Ar bottle
10:25	−55	406	382	370	295	"	~~80%~~	
10:47	−53	568	569	590	538	75%	85%	
11:14	−51	688	762	732	712	~~90~~	95%	
11:30	−50	797	864	825	801	80%	~~95~~%	
11:45	−49	795	795	801	799	"	80%	
12:05	−49				803	"	"	switch off, infiltrate in 60 secs
12:07	2525				755	0	0	
12:10	2232				662			
12:39	1468				280			
12:52	1312				195			pump out, open

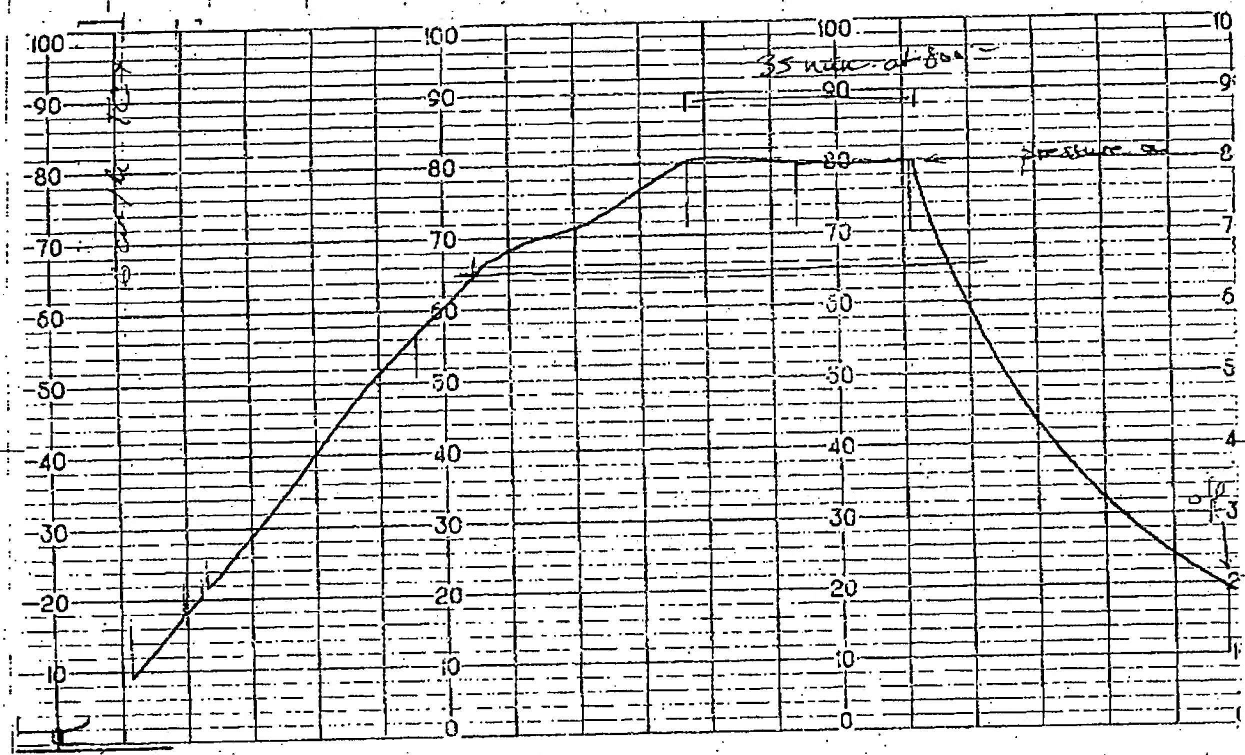
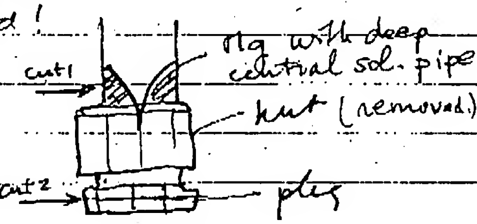


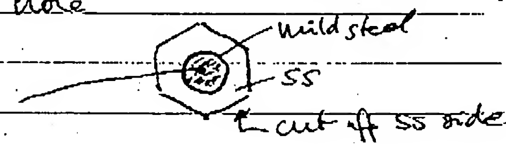
CHART NO. 860 195-6970-00

PRINTED IN U.S.A.

• Carbon crucible contains 4.0 g of ~~ctoo~~ coarse, grey dross could be Mg evaporated and condensed?

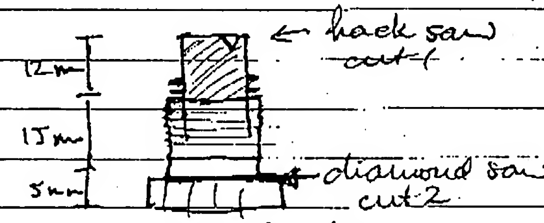
▲ cut large crucible → Mg has melted!
small oxide "hull" left in upper part, (very light) 0.43g. Some white deposit on top (MgO?). Cut
Cut 1: hack saw, see pipe hole
Cut 2: diamond saw.
looks infiltrated!





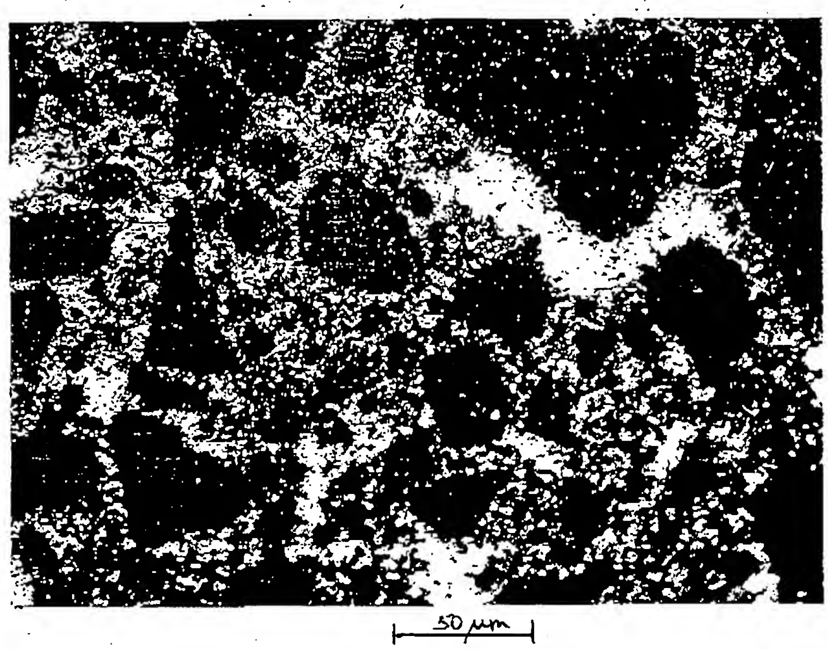
checked under microscope: diamond cut is good enough to show that the MgB_2 powders are infiltrated with Mg, forming a Mg/MgB_2 composite.
Took a polaroid, see next page.

▲ open small crucible
• cut 1: Mg to the rim, except small blowhole



• cut #2 shows only powder, no infiltration
• possible problem: 1) leak at nut
2) bad seal by liquid Mg (unlikely)
3) premature solidification (high surface area)

discard sample.



Mg: white
MgB_2: black

~1/3 Mg
2/3 MgB_2
should have continuous current path!

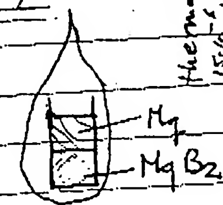
Polaroid: plug side of cut 2, very bottom of sample
sample Mg/MgB_2 (L) 40x objective (400x magnification)
- MgB_2 is agglomerated (size ~ 50-100 µm)
- Mg infiltrates as vein between agglomerates (veins: ~10-30 µm)
- many agglomerates are also filled with Mg (~1-5 µm)
- some show holes (pull out due to cutting?)

→ this is a fully infiltrated composite, with a few possible regions of porosity (maybe due to closed porosity in original powder or sintering)

TO DO: mount & polish this sample
machine steel off
measure conductivity vs T.

thermal exp: $15 \cdot 10^{-6}$, 1/8 ≈ 1.5%
OD: 15.875 mm
$\Delta OD = 0.24$ mm

next experiments (possible directions)



- does Mg wet MgB_2?
  use steel crucible to encapsulate
  MgB_2 + Mg
also may show enhanced sintering due to Mg vapors
(check density of green compact with He pycnometer

- react B fibers in capsule, then infiltrate
- " B powders " " "

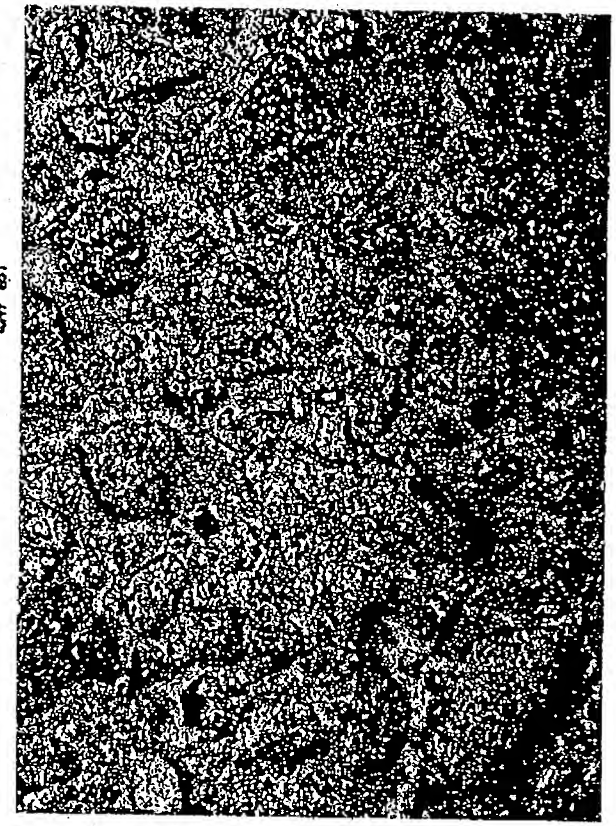
- infiltrate B fibers and then react in molten state
- " B powders " " " "

- infiltrate MgB_2 with Al } new crucibles
- " " " Cu } or BN coated steel

polishing same metallographic section (composite MMC
1. SiC paper with H_2O
2. 1μm diamond in oil (0.05 Al_2O_3 gums up matrix)
took 2 pictures (see next page)
- composite is successful: 1) full infiltration, no porosity
2) no reaction between Mg & MgB_2
3) no reaction of either phase w. crucible

Machine shop: asked for EDM of MMC!
welding of crucibles
Ordered: B powders, MgO powders, more steel

400x 50 µm



200x 100 µm

- large (~ 20-100 µm) MgB_2 } embedded in Mg matrix
- small (~ 2-20 µm) MgB_2

→ need to sieve

at the edge of composite, saw pure Mg annulus with some interdendritic shrinkage porosity. Not seen in composite

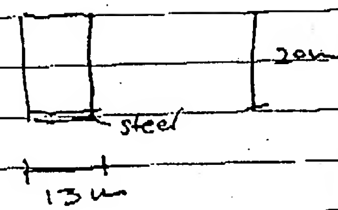
Metals which can be infiltrated in current machine

	Tm	IACS	
Cu	1083 (??)	100%	too hot?!
→ Ag	962	108%	
Al	662	65%	
Mg	650	39%	
Zn	420	28%	
Sn	232	16%	
Brass 260	955(l)	28%	
" 464	900	26%	
In	156	20%	
Pb	327	8%	
Cd	321	25%	
Au	1064	73%	too hot?!

Mutual solubilities in Mg-X systems

	in Mg	in X	intermetallics	eutectic
Mg-Mo	0	0	—	—
Mg-Nb	0	0	—	—
Mg-Co	0	0	$MgCo_2$	635
Mg-Cr	0	0	—	no phase diag
Mg-Fe	0	0	—	—
Mg-Be	0	0	1	
Mg-Nd	0	~2% (said)	4	548
Mg-Pd	0.23%	25at%	7	570
Mg-Pt	0	0	5	575
Mg-Ti	0.12%	1.5at%	~~20~~ —	651
Mg-Cu	0	6 at%	2	483
Mg-Au	0	huge	Many	575

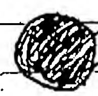
mass : 5.213 g

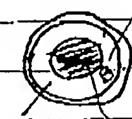


Received machined sample from machine shop. Machinist found composite to be much harder than stainless steel: boride seems well bonded to matrix!

· Side of cylinders show a few pure Mg regions (shiny) mark "B"
but mostly composite

· top part was faced →





· bottom part is ascut from diamond saw
→ need to cut again with diamond saw

The difference of color (gray for machined composite, black for cut composite) is probably due to smearing of Mg during lath machining.

· cut bottom slice (1.5 mm) with diamond saw (~2h!): other side looks nicely infiltrated, except for a large vein of pure Mg. Unlike the other side, it is not black (could be due to corrosion ...). Keep slice and mark it with a "B"

Actually, due to methanol! 1 min in it produces black color
it is probably etched by it!

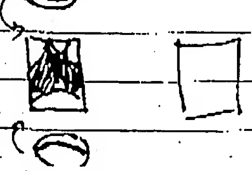
· cut upper slice ( )

· final dimensions: h: 17.044 mm
Ø: 12.748 mm r = 6.374 } ~~2.1753 cm³~~
m: 4.374 g

$\rho = 2.011$ g/cc

$f\,2.63 + (1-f)\,1.74 = 2.011$ $f\,(2.63-1.74) = 2.011-1.74 = 0.304$

30.5 vol% MgB_2



large regions of pure Mg, possibly displaced by falling chunks of Mg upon loading in crucible. In MMC region, volume fraction is larger.

PTO/SB/21 (09-04)
Approved for use through 07/31/2006. OMB 0651-0031
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

TRANSMITTAL FORM

(to be used for all correspondence after initial filing)

Application Number	10/680,639
Filing Date	October 7, 2003
First Named Inventor	David C. DUNAND
Art Unit	1742
Examiner Name	Ngoclan Thi Mai
Attorney Docket Number	6513-DIV
Total Number of Pages in This Submission	5

ENCLOSURES (*check all that apply*)

- ☐ Fee Transmittal Form
  - ☐ Fee Attached
- ☐ Amendment / Reply
  - ☐ After Final
  - ☐ Affidavits/declaration(s)
- ☐ Extension of Time Request
- ☐ Express Abandonment Request
- ☐ Information Disclosure Statement
- ☐ Certified Copy of Priority Document(s)
- ☐ Reply to Missing Parts/ Incomplete Application
  - ☐ Reply to Missing Parts under 37 CFR1.52 or 1.53
- ☐ Drawing(s)
- ☐ Licensing-related Papers
- ☐ Petition
- ☐ Petition to Convert to a Provisional Application
- ☐ Power of Attorney, Revocation Change of Correspondence Address
- ☐ Terminal Disclaimer
- ☐ Request for Refund
- ☐ CD, Number of CD(s) _____
  - ☐ Landscape Table on CD
- ☐ After Allowance Communication to TC
- ☐ Appeal Communication to Board of Appeals and Interferences
- ☐ Appeal Communication to TC (**Appeal Notice, Brief, Reply Brief**)
- ☐ Proprietary Information
- ☐ Status Letter
- ☒ Other Enclosure(s) (*please identify below*):

Supplemental Response;
Original, executed Declaration of David C. Dunand;
Return receipt postcard

Remarks

Authorization is hereby given to charge or credit Deposit Account No. 18-0882 for any fee deficiency or overpayment.

Rodney D. DeKruif

Rodney D. DeKruif

SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT

Firm	Reinhart Boerner Van Deuren s.c.		
Signature	Rodney D. DeKruif		
Printed Name	Rodney D. DeKruif		
Date	January 18, 2005	Reg. No.	35,853

CERTIFICATE OF TRANSMISSION/MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as Express Mail, No. EV 531802408 US, in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on the date shown below.

Signature	Rodney D. DeKruif		
Typed or printed name	Rodney D. DeKruif	Date	January 18, 2005

1 - 19 - 05

IFW

PATENT

I hereby certify that this paper or fee is being transmitted with sufficient postage as Express Mail, No. EV 531802408 US, in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on January 18, 2005.

Dated: January 18, 2005

BY: Rodney DeKruif
Rodney D. DeKruif

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:	David C. Dunand	
Serial No:	10/680,639	Attorney Docket No. 6513-DIV
Filed:	October 7, 2003	
For:	SUPERCONDUCTING Mg-MgB2 AND RELATED METAL COMPOSITES AND METHODS OF PREPARATION	

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

SUPPLEMENTAL RESPONSE

Dear Sir:

As a supplement to the Response filed on January 14, 2005, please enter and consider the original, executed Declaration of David C. Dunand, supporting patentability of the claimed invention. A copy of the same Declaration was previously filed.

Applicant believes no additional fees are required, but authorization is hereby granted to charge Deposit Account No. 18-0882 for any fee deficiency.

This application is now believed to believed to be in condition for allowance. Action consistent therewith is respectfully requested. Thank you for your time and consideration.

Respectfully submitted,

Rodney DeKruif

Rodney D. DeKruif
Attorney for Applicant
Reg. No. 35,853